

# इंटरनेट

# मानक

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IS 7918 (2001): Diethylene Glycol [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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भारतीय मानक  
डाईईथाईलीन गलाईकोल — विशिष्टि  
(पहला पुनरीक्षण)

*Indian Standard*  
DIETHYLENE GLYCOL — SPECIFICATION  
(*First Revision*)

ICS 71.080.10

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

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Price Group 4

## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Misc) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1975. The committee responsible for the preparation of this standard decided to revise the same in the light of the experience gained over the years so as to align the same with the latest development in technology and also on the quality of raw material presently available.

Diethylene glycol is used as a conditioning agent and lubricant for cotton, rayon and wool fibres to increase flexibility and improve elasticity. It is also used as a humectant for softening and controlling moisture. In the rubber industry, it is used in compounding to counteract the retarding effect of mineral fillers. Its other uses include gas dehydration, particularly natural gas, as a softening agent in glues and adhesives and in the extraction of aromatics. Considerable assistance has been derived from ASTM D2694: 1987 in preparation of this standard.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# Indian Standard

## DIETHYLENE GLYCOL — SPECIFICATION

( First Revision )

### 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for diethylene glycol for industrial use.

### 2 NORMATIVE REFERENCES

The following Indian Standards are necessary adjuncts to this standard. The standards contain provisions, which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water ( <i>third revision</i> )
2362 : 1993	Determination of water by the Karl Fischer method ( <i>first revision</i> )
4905 : 1968	Methods for random sampling
5298 : 1983	Method for determination of distillation range and of distillation yield ( <i>first revision</i> )
8768 : 1988	Method of measurement of colour in liquid chemical products in Hazen units ( <i>first revision</i> )

### 3 REQUIREMENTS

#### 3.1 Description

The material is hygroscopic. It shall be free from suspended matter and shall be miscible with water in all proportions.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the reference given under col 4 and 5 of Table 1.

#### 4 PRECAUTION IN HANDLING

The material shall be kept well protected from atmospheric humidity as it absorbs water vapour from the atmosphere.

### 5 PACKING AND MARKING

#### 5.1 Packing

The material shall be packed in well-closed containers preferably with replaceable closure.

5.2 The container shall be closed and shall bear legibly and indelibly following information:

- a) Name of the material;
- b) Net mass of the material in the container;
- c) Name of the manufacturer and his recognized trade mark, if any; and
- d) Batch number or lot number, in code or otherwise.

**Table 1 Requirements for Diethylene Glycol**  
(Clause 3.2)

SI No.	Characteristic	Requirements	Methods of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i)	Colour, Pt-Co scale, <i>Max</i>	15	—	8768
ii)	Relative density, 27°C/27°C	1.114 to 1.117	A	—
iii)	Distillation range;		B	—
	a) Initial boiling point 760 mm Hg, <i>Min</i>	240°C		
	b) Dry point 760 mm Hg, <i>Max</i>	252°C		
iv)	Water content, percent by mass, <i>Max</i>	0.2	C	—
v)	Acidity (as acetic acid) percent by mass, <i>Max</i>	0.01	D	—
vi)	Iron content ppm, <i>Max</i>	1.0	E	—
vii)	Monoethylene glycol content, percent by mass, <i>Max</i>	0.5	F	—
viii)	Triethylene glycol content, percent by mass, <i>Max</i>	1.0	F	—

**5.2.1 BIS Certification Marking**

The container may also be marked with the Standard Mark.

**5.2.1.1** The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standard Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

**6 SAMPLING**

The procedure for sampling and the criteria for conformity of the material shall be as prescribed in Annex G.

**7 QUALITY OF REAGENTS**

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

**ANNEX A**

[Table 1, *Sl No.* (ii)]

**DETERMINATION OF RELATIVE DENSITY****A-1 OUTLINE OF THE METHOD**

In this method, mass of equal volumes of the material and water are compared at 27°C.

**A-2 APPARATUS**

**A-2.1 Relative Density Bottle** — 25-ml capacity.

**A-2.2 Thermometer** — Any convenient thermometer of a suitable range with 0.1°C or 0.2°C subdivisions.

**A-2.3 Water-Bath** — capable of being maintained at 27.0 ± 0.2°C.

**A-3 PROCEDURE**

Clean and dry the relative density bottle. Weigh and fill with recently boiled and cooled water at 27°C. Fill to overflowing by holding relative density bottle on its side in such a manner as to prevent entrapment of air bubbles.

Insert the stopper and immerse in the water-bath maintained at 27.0 ± 0.2 °C. Keep the entire bulb covered with water and hold at that temperature for 30 minutes. Carefully remove any water, which has exuded from the capillary opening. Remove from the bath, wipe completely, dry and weigh. Again clean and dry the relative density bottle. Using the material under test, proceed exactly as in the case of water and weigh the bottle with the material.

**A-4 CALCULATION**

$$\text{Relative density at } 27^{\circ}\text{C}/27^{\circ}\text{C} = \frac{A - B}{C - B}$$

where

$A$  = mass in g of the relative density bottle with the material,

$B$  = mass in g of the relative density bottle, and

$C$  = mass in g of the relative density bottle with water.

**ANNEX B**

[Table 1, *Sl No.* (iii)]

**DETERMINATION OF DISTILLATION RANGE****B-1 PROCEDURE**

Determine the distillation range by the procedure as prescribed in IS 5298 applying following corrections.

**B-2 CORRECTION FOR THERMOMETER READING****B-2.1 Error in Scale**

In all the thermometer readings, make the corrections as indicated on the calibration certificate of the instrument.

**B-3 CORRECTION FOR BAROMETRIC PRESSURE**

If the barometric pressure prevailing during the determination is 760 mm Hg, no correction need be applied to the specified temperature and the thermometer reading may be used as such. If however the prevailing barometric pressure deviates from 760 mm Hg, the specified temperature shall also be corrected as follows:

- a) For every 10 mm above 760 mm Hg, subtract 0.5°C from the specified temperature; and

- b) For every 10 mm below 760 mm Hg, add 0.5°C to the specified temperature.

NOTE — These corrections are valid only for pressure above 700 mm Hg.

## ANNEX C

[Table 1, Sl No. (iv)]

### DETERMINATION OF WATER CONTENT

#### C-1 GENERAL

Water content is determined by the Karl Fischer method.

#### C-2 PROCEDURE

Take about 20 g of the material, weigh accurately and determine the water content by the procedure given in IS 2362.

## ANNEX D

[Table 1, Sl. No. (v)]

### DETERMINATION OF ACIDITY AS ACETIC ACID

#### D-1 OUTLINE OF THE METHOD

The material is titrated with standard sodium hydroxide solution to the phenolphthalein end point and from the volume of standard sodium hydroxide solution used, acidity is calculated as acetic acid.

#### D-2 REAGENTS

**D-2.1 Sodium Hydroxide Solution** — Approximately 0.1 N.

**D-2.2 Phenolphthalein Indicator** — 0.5 percent (*m/m*) in *iso*-propyl alcohol and make it faintly pink by the addition of dilute sodium hydroxide solution.

#### D-3 PROCEDURE

Weigh accurately about 100 g of the material to the nearest 0.1 g in a 500-ml conical flask. Pour about 100 ml of freshly boiled and cooled distilled water and few drops of phenolphthalein indicator. Mix-well and note the colour of the solution. A pink colouration indi-

cates the presence of alkalinity. If the solution is colourless, titrate with standard sodium hydroxide solution until a pale pink colour persists for more than 15 seconds.

Take 100 ml of distilled water in another 500-ml conical flask and carry out a blank test.

#### D-4 CALCULATION

$$\text{Acidity (as acetic acid), percent by mass} = \frac{(V_s - V_b) \times 6 \times N}{M}$$

where

$V_s$  = Volume in ml of standard sodium hydroxide solution used in the test with the material,

$V_b$  = Volume in ml of standard sodium hydroxide solution used in the blank test,

$N$  = Normality of standard sodium hydroxide solution, and

$M$  = Mass in g of the material taken for the test.

## ANNEX E

[Table 1, Sl No. (vi)]

## DETERMINATION OF IRON

## E-1 GENERAL

Two methods for determination of iron content have been prescribed, namely.

- a) Ortho-phenanthroline method, and
- b) Ammonium thiocyanate method.

## E-2 ORTHO-PHENANTHROLINE METHOD

## E-2.1 Outline of the Method

The sample is diluted with water and the iron determined photometrically at approximately 510 nm by the ortho-phenanthroline method without a preliminary ashing. In order to overcome the depression of the ferrous-ortho-phenanthroline complex a large excess of hydroxylamine hydrochloride and ortho-phenanthroline solutions are added.

## E-2.2 Apparatus

**E-2.2.1 Spectrophotometer** — capable of measuring light absorption at approximately 510 nm.

**E-2.2.2 Absorption Cell** — 4-cm light path.

## E-2.3 Reagents

**E-2.3.1 Hydroxylamine Hydrochloride Solution** — 300 g/l.

**E-2.3.2 Iron, Standard Solution** (1 ml = 0.005 mg Fe) — Weigh exactly 0.702 2 g of ferrous ammonium sulfate hexahydrate [ $\text{FeSO}_4(\text{NH}_4)_2 \cdot 6\text{H}_2\text{O}$ ] minimum purity 99.5 percent, dissolving in 500 ml of water containing 20 ml of sulphuric acid ( $\text{H}_2\text{SO}_4$ , Sp. Gr. 1.84) and diluting to 1 000 ml with water. Dilute 50 ml of this solution to 1 000 ml for 0.005 mg/l.

**E-2.3.3 Ortho-phenanthroline Solution** — 3 g/l.

Dissolve 0.75 g of 1,10 phenanthroline monohydrate in 25 ml of iron free ethanol and dilute to 250 ml with water.

## E-2.4 Procedure

## E-2.4.1 Preparation of Calibration Curve

Prepare a series of at least five standards by adding 1.0 ml to 12.0 ml of standard iron solution (1 ml = 0.005 mg Fe) to 100 ml glass-stoppered cylinders. Make up to 80 ml with water. Add 80 ml water to another glass-stoppered cylinder as a blank. Add 5 ml of hydroxylamine hydrochloride solution, to each flask and make up to 100 ml with ortho-phenanthroline solution and mix. Allow to stand for 5 minutes. Obtain the absorbance of

each standard, corrected for the blank. Prepare a calibration curve by plotting the absorbance of the standard iron solutions in 5 cm cells against the milligrams of iron per 100 ml of solution.

## E-2.4.2 Analysis of Sample

Weigh about 80 g of sample nearest to 0.1 g in a 100 ml glass stoppered cylinder. Make up to 80 ml with water. Add 80 ml of water to another glass stoppered cylinder as blank. Add 5 ml of hydroxylamine hydrochloride to each cylinder and make up to 100 ml mark with ortho-phenanthroline solution and mix. Allow to stand for 5 minutes and measure the absorbance using 5 cm cells at 510 nm corrected for blank. Read mg of iron from graph.

## E-2.5 Calculation

Calculate iron present, in parts per million, as follows:

$$\text{Iron, ppm} = (W/S) \times 1\,000$$

where

$W$  = iron found, mg; and

$S$  = sample used, g.

## E-3 AMMONIUM THIOCYANATE METHOD

## E-3.1 Outline of the Method

The colour produced by a known quantity of the material with ammonium thiocyanate is compared with a control containing known quantity of iron.

## E-3.2 APPARATUS

**E-3.2.1 Nessler Cylinders** — 50 ml capacity.

## E-3.3 Reagents

**E-3.3.1 Hydrochloric Acid** — 1:1 (v/v).

**E-3.3.2 Potassium Permanganate Solution** — 0.1 N approximately.

**E-3.3.3 Ammonium Thiocyanate Solution** — 60 percent (m/v).

**E-3.3.4 Mixture of Amyl Alcohol and Amyl Acetate** — 1:1 (v/v).

## E-3.4 Standard Iron Solution

## E-3.4.1 Standard Iron Solution A

Dissolve 0.702 2 g of ferrous ammonium sulphate [ $\text{FeSO}_4(\text{NH}_4)_2 \cdot 6\text{H}_2\text{O}$ ] in water in a one-litre volumetric flask. Add 4 ml of concentrated sulphuric acid and

make up the volume to the mark with water. One millilitre of this solution contains 0.1 mg of iron (as Fe).

#### E-3.4.2 Standard Iron Solution B

Take 100 ml of standard iron solution A and dilute to 1 000 ml with water in a one-litre volumetric flask. One millilitre of this solution contains 0.01 mg of iron (as Fe). This solution shall be prepared fresh.

#### E-3.4 Procedure

Accurately weigh 50 g of the sample in a platinum or silica dish (100 ml capacity). Place on a sand-bath and carefully evaporate to dryness. Cool, add 2 ml of hydrochloric acid and 25 ml of water and heat to facilitate dissolution and cool. Add one drop of potassium

permanganate solution and mix thoroughly. Add 5 ml of ammonium thiocyanate solution and 10 ml of amyl alcohol and amyl acetate mixture. Make up to 50 ml, shake vigorously and allow the layers to separate. Compare the intensity of any red colour produced in the upper layer with a control test carried out in another Nessler cylinder in the same manner using 5 ml of standard iron solution B in the control test.

#### E-3.5 INTERPRETATION OF RESULTS

The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the respective control test.

## ANNEX F

[Table 1, *Sl No. (vii) and (viii)*]

### DETERMINATION OF MONOETHYLENE AND TRIETHYLENE GLYCOL

#### F-1 OUTLINE OF THE METHOD

A small volume of diethylene glycol is injected into the gaschromatograph containing a packed column with Carbowax 20 M as stationary phase on chromosorb G HP as solid support.

Quantitative results are obtained by using suitable electronic integrator and external standardization technique, which gives direct concentration of each impurity along with the chromatogram, area, retention time, etc.

#### F-2 APPARATUS

**F-2.1 Gas Chromatograph** — Any instrument having a thermal flame ionization detector that may be operated at the conditions given below:

Detector	: Flame ionization
Column	: Stainless steel
Length	: 110 cm
I.D./O.D.	: 2 mm / 1/8 inch
Stationary phase	: Carbowax 20 M
Solid support	: Chromosorb G HP 100/ 120 mesh
Injector type	: Packed with glass wool
Temperatures	
Injection Port	: 250 °C
Detector	: 250 °C
Oven initial	: 150 °C
Initial time	: 2 minutes
Programming rate	: 5 °C/minute
Final temperature	: 200 °C

Final time	: 15 minutes
Carrier gas	: Nitrogen
FID hydrogen	: 25 ml/minute
Air	: 250 ml/minute
Sample size	: 3.0 microlitre diluted with iso-propyl alcohol

NOTE — The above gas chromatographic conditions are only suggestive, however, other gas chromatographs with different columns may be used provided standardization/calibrations are done after setting up chromatographic conditions for the required resolution.

#### F-3 REAGENTS

Nitrogen Chromatographic Grade

Pure Compound for Calibration — monoethylene glycol and triethylene glycol Isopropyl alcohol

#### F-4 PROCEDURE

Prepare a synthetic mixture containing known weight percentage of MEG and TEG in pure DEG. Dilute synthetic mixture 1:1 with iso-propyl alcohol. Set the chromatograph and the electronic integrator as required, and inject 3 microlitre of diluted synthetic mixture. Allow the chromatogram to develop. At the end of the run stop the integrator. Calculate factors as follows:

$$FM = \frac{\% \text{ MEG obtained by GC}}{\text{Actual \% MEG in synthetic mixture}}$$

$$FT = \frac{\% \text{ TEG obtained by GC}}{\text{Actual \% TEG in synthetic mixture}}$$

where

$FM$  = Factor for MEG

$FT$  = Factor for TEG

(Run the calibration mixture in duplicate and take the average factor for calculation to estimate MEG/TEG in sample).

Inject sample following the same procedure/conditions used for synthetic mixture. Calculate % concentration of components in DEG as follows.

#### F-5 CALCULATION

% MEG = % MEG found by GC in sample  $\times$  FM

% TEG = % TEG found by GC in sample  $\times$  FT

## ANNEX G

(Clause 6)

### SAMPLING

#### G-1 GENERAL REQUIREMENTS

In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**G-1.1** Sample shall not be taken in an exposed place.

**G-1.2** The sampling instruments shall be clean and dry and shall be made of low or reduced spark generating material.

**G-1.3** The samples, the material being sampled, the sampling instrument and the containers for samples shall be protected from adventitious contamination.

**G-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by shaking or stirring or both, or by rolling, so as to bring all portions into uniform distribution.

**G-1.5** The samples shall be placed in suitable, clean, dry and airtight glass containers preferably of amber or blue colour.

**G-1.6** The sample containers shall be of such a size that they are almost, but not completely, filled by the sample.

**G-1.7** Rubber stoppers or composition corks shall not be used for closing the sample bottles.

**G-1.8** Sealing wax or other plastic material, of use shall be applied in such a way that it does not contaminate the sample.

**G-1.9** Each sample container shall be protected by cover of oil proof paper, metal foil, viscous or other suitable impervious material over the stopper to keep away moisture and dust from the mouth of the bottle and to protect it while being handled.

**G-1.10** Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling, such as the date of sampling, the year of manufacture, the batch number, the name of the

sampler, etc. Particular care shall be taken to ensure that sealing methods do not contaminate the sample.

#### G-2 SAMPLING INSTRUMENT

The following forms of sampling instruments may be used:

- Sampling bottle or can, for taking samples from various depths of large tanks; and
- Sampling tube.

##### G-2.1 Sampling Bottles or Can

It consists of weighed bottle or metal container with removable stopper of top, to which is attached a light chain (see Fig. 1). The bottle or can is fastened to a suitable pole. For taking a sample, it is lowered in the tank to the required depth and the stopper or top is removed by means of the chain for filling the container.

##### G-2.2 Sampling Tube

It is made of metal or thick glass and is about 20 to 40 mm in diameter and 400 to 800 mm in length (see Fig. 2) depending on the type of container from where the sample is to be drawn.

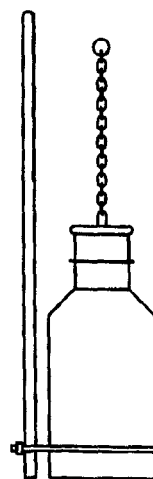
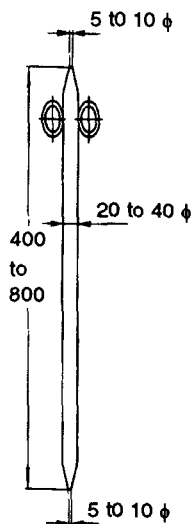


FIG. 1 SAMPLING BOTTLE OR CAN



All dimensions in millimetres.

FIG. 2 SAMPLING TUBE

The ends are conical and reach 5 to 10 mm diameter at the tips. Handling is facilitated by two rings at the upper end. For taking a sample, the apparatus is first closed at the top with thumb or a stopper and lowered until the desired depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn.

### G-3 SCALE OF SAMPLING

#### G-3.1 Lot

In any consignment, all the containers of the same size and drawn from same batch of manufacture shall constitute a lot.

**G-3.2** Tests should be conducted on each lot separately for ascertaining the conformity to the requirements of this specification. The number of containers to be chosen at random from the lot for this purpose shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 2.

**G-3.3** The containers shall be selected from the lot at random and in order to ensure the randomness of

selection, the method given in IS 4905 may be followed.

**Table 2 Scale of Sampling**

(Clause-G-3.2)

Number of Containers in a Lot (1)	Number of Container to be Selected (2)
Up to 25	3
26 to 100	4
101 to 150	5
151 and above	7

### G-4 PREPARATION OF THE TEST SAMPLE

**G-4.1** From each of the containers selected according to G-3.2 and G-3.3 small portions of the material shall be drawn with the help of the sampling instrument from several positions at the upper, middle and bottom layers. These portions shall be mixed together so as to yield a quantity of material not less than 1 000 ml representative of the container.

**G-4.2** From each of these individual samples, an equal quantity of material shall be taken and mixed thoroughly to form a composite sample of about 1 000 ml. The composite test sample shall be divided into three equal parts, one for the purchaser, another for supplier and third to be used as a referee sample.

**G-4.3** The parts of the composite test sample, obtained according to G-4.2 shall be transferred to separate containers and shall be sealed and marked with full identification particulars given in G-1.10.

**G-4.4** The referee test sample shall bear the seals of both the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and supplier to be used in case of any dispute between the two.

### G-5 CRITERIA FOR CONFORMITY

**G-5.1** The material shall be declared as conforming to the requirements of this specification if the composite sample conforms to all the specified requirements.

**Bureau of Indian Standards**

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**Review of Indian Standards**

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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**Amendments Issued Since Publication**

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